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ENGINEERING AND DEVELOPMENT SUPPORT OF GENERAL DECON TECHNOLOGY FOR THE DARCOM INSTALLATION RESTORATION PROGRAM

Task 8. Identification of Recovered Organics from Supercritical Fluid Process to Regenerate Carbon

FINAL REPORT

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William E. Jones
Debra A. Price
Judith F. Kitchens

August 1981

Submitted to:

Commander
U.S. Army Toxic and Hazardous Materials Agency
Aberdeen Proving Ground (Edgewood Area), Maryland 21010

Ned Colburn
Project Officer

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ATLANTIC RESEARCH CORPORATION
5390 Cherokee Avenue
Alexandria, Virginia 22314

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A. Objective

The purpose of this task was to qualitatively identify the contaminants of Rocky Mountain Arsenal groundwater by GC/MS and compared the results to a GC/MS scan of the supercritical fluid extract of carbon provided by A.D. Litle. In addition to the qualitative analysis, a semi-quantitative analysis of the DIMP levels in both groundwater and carbon extract was performed.

B. Qualitative Analysis of RMA Groundwater

A 200 ml sample of the RMA groundwater was extracted with two 5 ml of ethyl ether. The extract was further concentrated to approximately 0.1 ml in a micro "Kuderna Danish" evaporator for GC/MS analysis. The chromatographic conditions were as follows:

Column:	2% Dexsil 300 on Anachrom Q
Oven Temperature:	140 - 200°C @ 15°C/minute
Injector Temperature:	200°C

Mass scanning was from 40 - 550 AMU at 8 samples per 0.1 AMU. An electron multiplier potential of 2,000 volts was employed.

The chromatogram of the groundwater extract gave two main peaks. The first gave a mass spectrum with major ions at 97 and 123 AMU. Comparison of this spectrum to that found for an authentic sample of DIMP established the identity of the peak. The second peak was tentatively identified as 2,6-di(τ-butyl)4-methylphenol by a manual search of the EPA/NIH Mass Spectral Data Base.

C. Analysis of A.D. Little U-Tube

As received from A.D. Little, the U-tube containing the supercritical extract from the carbon was wrapped in aluminum foil. Removal of the foil exposed a U-tube capped with teflon stopper. The tube had broken where the stopper was inserted but there was no evidence of loss of any of the tube's contents. The

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Eric Hangeland
CETHA-TS-D
APG, MD 21010-5401
NWW 1-29-92

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tube contained about 4 - 5 ml of what appeared to be water. The tube contents were extracted by addition of two separate 5 ml portions of ether. The tube was recapped each time and shaken well before pouring out the contents. The combined extracts were separated from the water phase and injected directly without concentration. The U-tube extract showed the presence of only DIMP.

D. Semi-Quantitative Determination of DIMP Content in the RMA Ground Water and U-Tube

A second 100 ml sample of RMA groundwater was extracted twice with chloroform, the total extract amounting to 2 ml. Analysis was performed on a Hewlett Packard 5880 gas chromatograph equipped with a nitrogen phosphorous detector with the following conditions:

Column:	1.5% OV-17/1.95% OV-210	
Temperature:	Oven	130°C
	Injector	300°C
	Detector	320°C
Carrier:	N_2 @ 28 cc/minute	
Auxillary Gas:	H_2 @ 4 cc/minute	
	Air @ 90 cc/minute	
Sensitivity:	532 AU/ng (based on a 20.3 ng injection)	
Retention Time:	varies with sample size (0.8 - 1.5 minutes)	

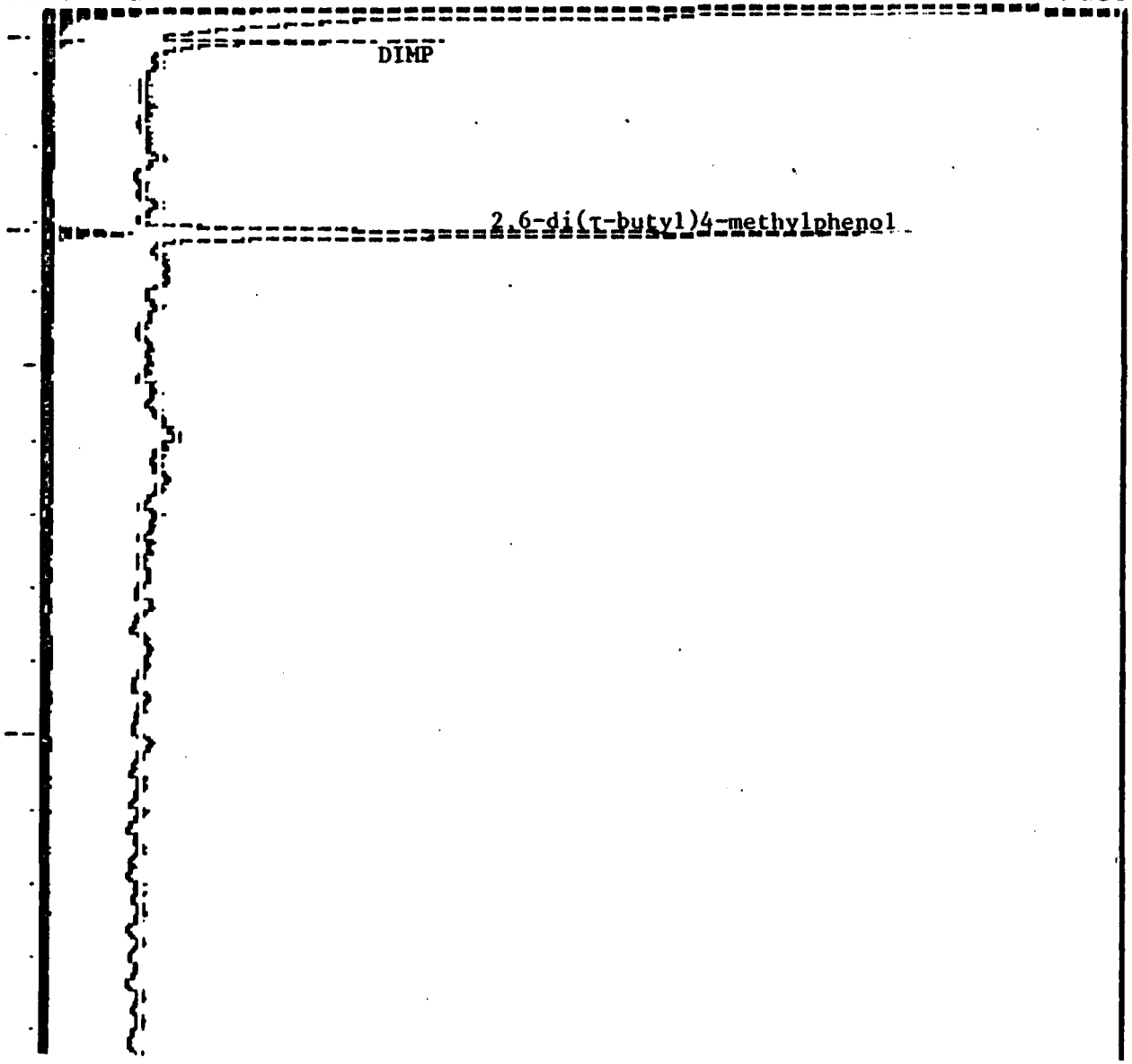
The RMA groundwater contained 1.55 mg/L of DIMP. The U-tube extract contained 31.47 mg/L of DIMP or a total 0.315 mg of DIMP. The efficiency of the supercritical carbon extraction can be determined if the amount of groundwater run through the carbon is known.

SAMPLE NAME WELL WATER EXTRACT (GC/MS)

OPERATOR

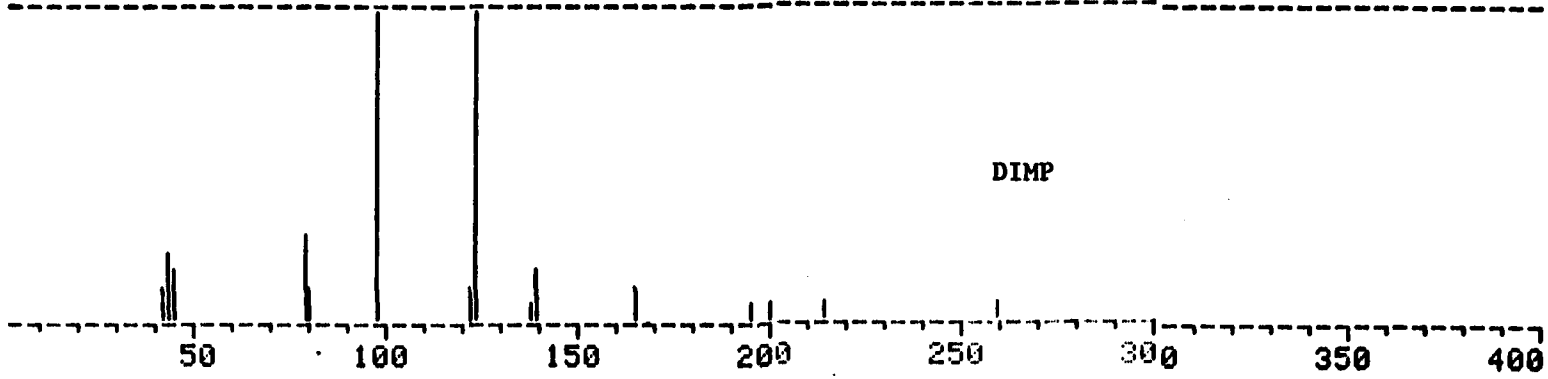
TOTAL ABUNDANCE FROM 40 TO 450 amu
Full Scale= 9920

ION 269.0
Full Scale= 150

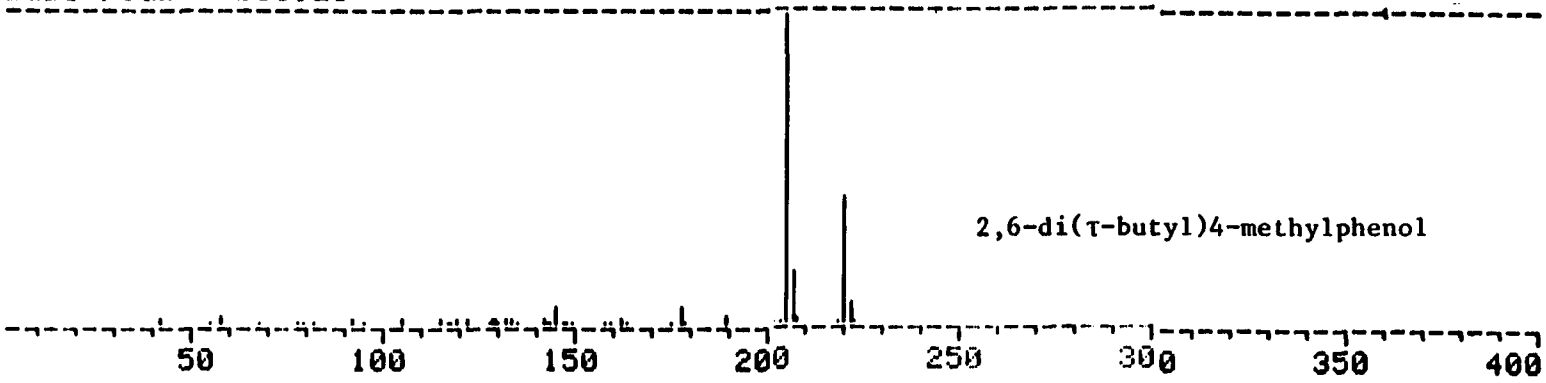


GROUNDWATER EXTRACT - MASS SPECTRA

** Spectrum # 84 ** Sample # 1 Retention Time = 0.4 minutes
Scanned from 40 to 450 amu Number of Peaks Detected = 15
File type = linear
Base Peak = 96.95

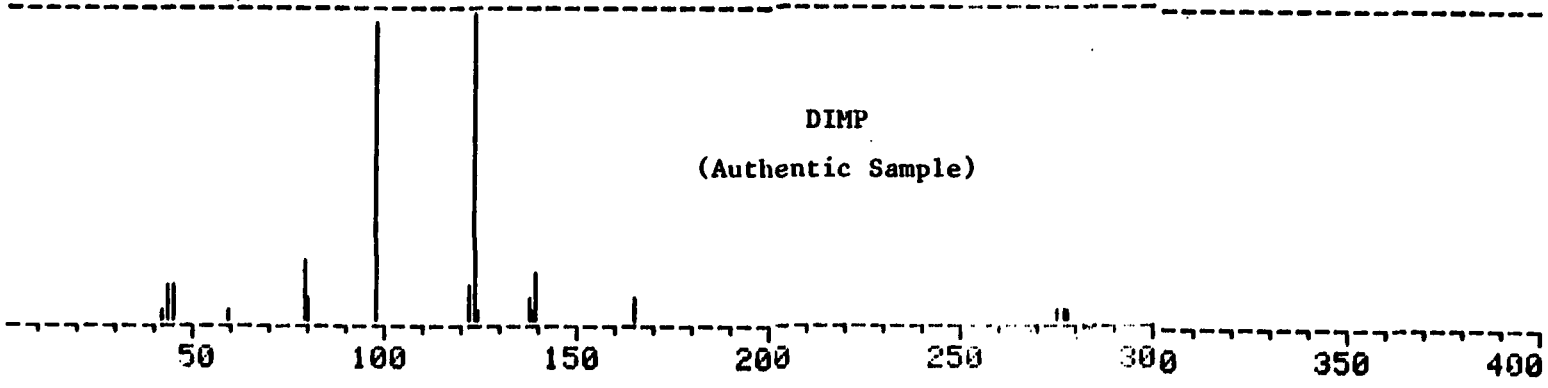


** Spectrum # 85 ** Sample # 1 Retention Time = 3.1 minutes
Scanned from 40 to 450 amu Number of Peaks Detected = 83
File type = linear
Base Peak = 205.20

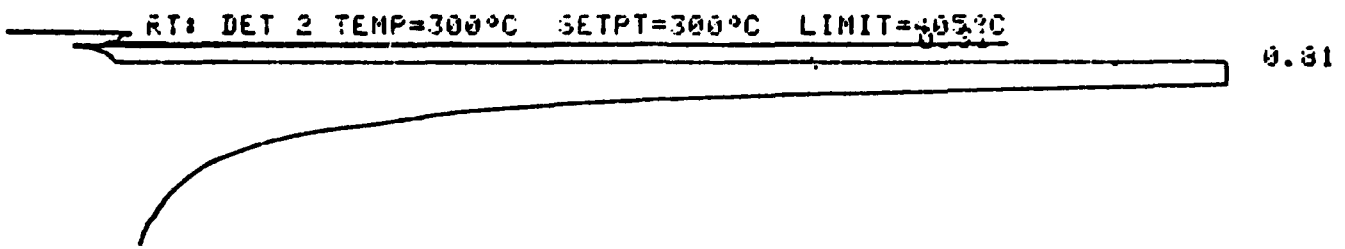


DIMP - Mass Spectra

** Spectrum # 28 ** Sample # 1 Retention Time = 0.7 minutes
Scanned from 40 to 550 amu Number of Peaks Detected = 17
File type = linear
Base Peak = 123.00



Chromatograph of Groundwater Extract

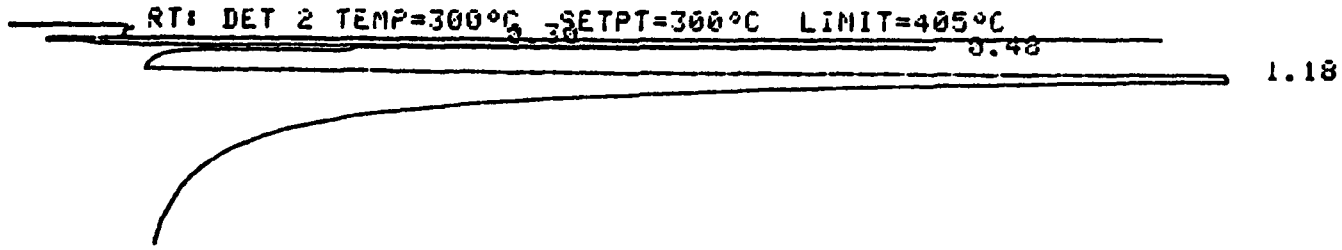


[hp] 5880A MANUAL INJECTION @ 14:30 AUG 7, 1981
AREA %

RT	AREA	TYPE	WIDTH	HEIGHT	BASELINE	AREA %
0.00			BASELINE @ START RUN = 48.28			
0.00			THRESHOLD @ START RUN = 1			
0.00			PEAK WIDTH @ START RUN = 0.08			
0.31	510.85	BP	0.024	334.27	33.48	0.646
0.81	78542.80	PB	*0.28 *	4453.97	28.75	99.354

TOTAL AREA = 79053.70
MULTIPLIER = 1

Chromatograph of U-Tube Extract



[illegible] MANUAL INJECTION @ 14:17 AUG 7, 1981
AREA %

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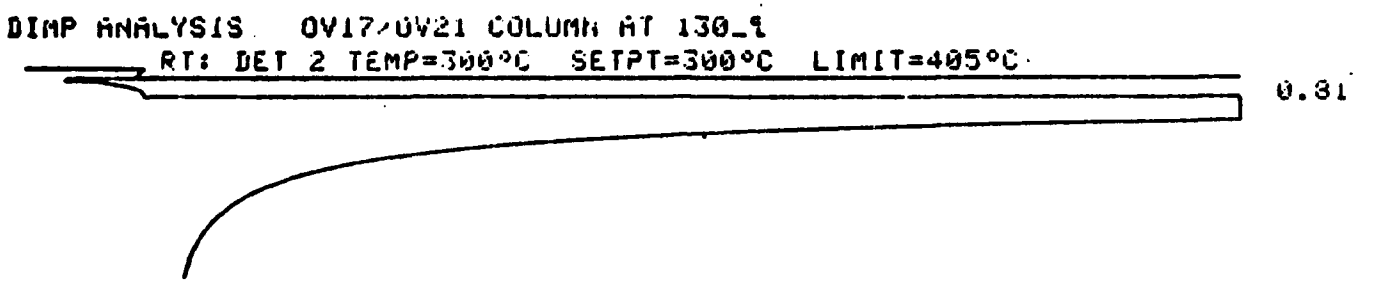
0.00			BASELINE @ START RUN = 37.61			
0.00			THRESHOLD @ START RUN = 1			
0.00			PEAK WIDTH @ START RUN = 0.08			
0.30	135.15	VP	0.012	175.84	5.47	0.381
0.48	2876.82	PH	*------*	376.91	0.94	8.115
1.18	32439.80	HH	------*	646.03	0.94	91.504

TOTAL AREA = 35451.80
MULTIPLIER = 1

Chromatograph of DIMP 77 ppm (SARM)



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[redacted] ANNUAL INJECTION @ 13:41 AUG 7, 1981

RT	AREA	TYPE	WIDTH	HEIGHT	BASELINE	AREA %
0.00			BASELINE @ START RUN = 35.65			
0.00			THRESHOLD @ START RUN = 1			
0.00			PEAK WIDTH @ START RUN = 0.08			
0.81	80947.10	PH	*0.36 *	3519.01	0.61	100.000

TOTAL AREA = 80947.10
MULTIPLIER = 1